

\* NOTICES \*

JPO and INPIT are not responsible for any damages caused by the use of this translation.

1.This document has been translated by computer. So the translation may not reflect the original precisely.

2.\*\*\* shows the word which can not be translated.

3.In the drawings, any words are not translated.

**CLAIMS**

(57) [Claim(s)]

[Claim 1]A constituent for metal finishing consisting of an organic silicon compound, an epoxy resin, polyvinyl butyral resin, and an organic solvent dispersibility silica particle which have an amino alkyl group and alkoxy silyl groups.

[Translation done.]

## \* NOTICES \*

JP0 and INPI are not responsible for any damages caused by the use of this translation.

1. This document has been translated by computer. So the translation may not reflect the original precisely.
- 2.\*\*\* shows the word which can not be translated.
3. In the drawings, any words are not translated.

## DETAILED DESCRIPTION

[Detailed Description of the Invention]

(b) The purpose of invention "Field of the Invention"

This invention relates to the constituent for metal finishing, and in more detail, when it applies to galvanization or a zinc system alloy plating steel plate etc. in which chemical conversion was performed, it has film adhesion, damage resistance, and rust prevention with a good rear spring supporter at a long period of time, and it is related with the composition for surface treatment which forms the effective coat also as a paint ground, and can use widely by the various fields of industry which use metal, such as machine industry, electrical machine industry, and the auto industry.

## "PRIOR ART"

From the former, many processing agent and paints are used in order to protect the surface of a galvanized steel sheet or a zinc alloy galvanized steel sheet from various corrosive environment. Phosphate treating or chromate treatment is common also in it. However, if independent, there was only a primary rust prevention role, and even when [ its ] rust occurred for a short time and it was premised on finishing, problems, such as carrying out rusting, were in the storage time to finishing. In recent years, the steel plate which made resin membrane form on the zinc system galvanized steel sheet which performed chemical conversion, such as chromate treatment and phosphate treating, for the purpose of improvement in rust prevention is proposed and put in practical use. Endurance, such as the damage resistance at the time of handling, anti-fingerprint property, the adhesion at the time of a fabricating operation, crack-proof nature and paintwork at the time of finish coating, adhesion and rust prevention, and a water resisting property, etc. are demanded of this steel plate.

"The problem which invention tends to solve"

the metal antirust which uses as the main ingredients the organic silicon compound, the epoxy resin, and thermoplastics which have an amino alkyl group and alkoxy silyl groups previously, such as this invention person, — business — the constituent was proposed (JP.60-238372A).

When this constituent is applied to a steel plate, it has the outstanding rust prevention, but the rust prevention in processing sections which improved more to this constituent, such as bending and a diaphragm, and the adhesion durability at the time of finish coating are searched for.

(\*\*) Composition of invention "means for solving a problem"

the metal antirust in which the rust prevention where this invention persons satisfied the above-mentioned demand, and which was excellent is shown — business, as a result of inquiring wholeheartedly in order to ask for a constituent. The organic silicon compound which has an amino alkyl group and alkoxy silyl groups, the metal antirust which demonstrates the processing section rust prevention where the constituent which distributed and mixed the silica particle filled the above-mentioned request to the constituent which consists of an epoxy resin and polyvinyl butyral resin, and which was excellent in it, and finish coating adhesion — business — it found out that it could become a constituent and this invention was completed. That is, this invention relates to the constituent for metal finishing consisting of the organic silicon compound, the epoxy resin, polyvinyl butyral resin, and the organic solvent dispersibility silica particle which have an amino alkyl group and alkoxy silyl groups.

Constituent features of this invention is explained in full detail below.

O Organic silicon compound The organic silicon compound used by this invention includes what it has both an amino alkyl group and alkoxy silyl groups, and the thing of a description is similarly used for above-mentioned published unexamined application, and is widely known as a silane coupling agent. Some which have two or more alkoxy silyl groups have an effect especially in improvement in rust prevention, take to this invention, and are preferred.

[http://www4.ipdl.inpit.go.jp/cgi-bin/tran\\_web.cgi\\_ejie?atw\\_u=http%3A%2F%2Fwww4.ipdl.inpit.go...](http://www4.ipdl.inpit.go.jp/cgi-bin/tran_web.cgi_ejie?atw_u=http%3A%2F%2Fwww4.ipdl.inpit.go...) 2010/10/22

O Epoxy resin What used as the base the bisphenol A in which the epoxy resin in which a thing given [ as an epoxy resin used by this invention ] in above-mentioned published unexamined application is similarly used, and has two or more hydroxyl groups in one molecule in this invention is used desirable especially general-purpose is preferred.

As a commercial item of a bisphenol A type epoxy resin, Epicoat 1001, 1004, 1007, and 1009 (above, product made from Oil recovery Shell Epoxy) etc. are mentioned, for example. Brominated epoxy resin 1045 by which some hydrogen atoms of the benzene ring of bisphenol A type epoxy resin were replaced with bromine, for example, Epicoat, and Epicoat YL903 (above, product made from Oil recovery Shell Epoxy) etc. is mentioned.

As an epoxy resin of commercial items other than a bisphenol A type epoxy resin, Phenoxyl resin (made in Union Carbide Corporation), for example, PKHH etc., such as the dimer acid system glycidyl ester typed epoxy resin 871 (product made from Oil recovery Shell Epoxy), for example, Epicoat etc., is mentioned. The Reason the epoxy resin which has two or more hydroxyl groups in one molecule in this invention,

especially a bisphenol A type epoxy resin are preferred reacts very easily [ the oxirane ring of the amino alkyl group of an organic silicon compound, and an epoxy resin ]. When it has two or more hydroxyl groups compared with coat formation taking a long time late to reaction velocity when the number of the hydroxyl groups in one molecule is less than two pieces, the hydroxyl group of an epoxy resin is considered because it has contributed to the reaction with an organic silicon compound greatly, but it is because a tough coat is formed in the inside of a short time.

O Polyvinyl butyral resin With polyvinyl butyral resin in this invention constituent. What butyral-ized with butylaldehyde the polyvinyl alcohol produced by saponifying polyvinyl acetate is said, and, generally the thing of various physical properties is obtained with the presentation, a degree of polymerization, etc. by the copolymer of vinyl butyral, vinyl acetate, and vinyl alcohol. The process and character of polyvinyl butyral resin are indicated to Encyclopedia of polymer science & technology vol.14 p.208-239 (John Wiley & Sons).

As a commercial item of polyvinyl butyral resin, S lek BL1, S lek BL2, S lek BMS, S lek BLS, and S lek BX1 (above, Sekisui Chemical Co., Ltd. make) etc. is mentioned, and it is used for this invention, for example.

It is not limited to the kind, the hydroxyl group and organic silicon compound which are contained in resin react, and polyvinyl butyral resin in this invention constituent is considered to contribute to formation of a tough coat.

O Silica particle The silica particle used by this invention. When applying this invention constituent to a surface of metal, in order to generally use it as an organic solvent solution. If it may distribute to an organic solvent, are not limited in particular, but as a silica particle with a particle diameter [ the field of organic solvent dispersibility to ] of 0.01-1 micrometer, or a composition metal ingredient — silicon — more than 50 mol % — the particles of composite metal oxides, such as silicon to contain, zirconia, titanium, aluminum, boron, or compound hydroxide are preferred.

As such fine-powder-form silica, in the first place [ \*\*\* ] The particle-like silicic acid anhydride as a commercial item. Namely, what is called fumed silica and the hydrophobic silica (hydrophobic silica) which processed fumed silica by Silang or a polysiloxane can be mentioned. The details of that are indicated to Encyclopedia of Chemical Technology (Secand Edition) Vol.18 p.61-72. As a concrete commercial item, Aerosil 200, Aerosil 300, Aerosil R972, Aerosil R810 (above, product made from Japanese Aerosil), etc. are mentioned, for example.

A metal alkoxide or a metal coordination compound is used as a raw material, the silica particle (henceforth compound silica) obtained by the hydrolysis using a basic catalyst, a polycondensation reaction, and what is called sol gel process is mentioned to the second, and it is a desirable silica particle from fields, such as reactivity with resin, and the rust prevention of a processing section.

It will be as follows if it explains in full detail about the process of the compound silica obtained by the sol gel process desirable for this invention.

The solution of a kind or two sorts or more of alkoxy silane compounds Under existence of a basic catalyst, 0-200 \*\* 0.5-20 mol is hydrolysis and a method of carrying out a polycondensation reaction at 20-150 \*\* more preferably by 0.75-10-mol water to 1 mol of alkoxy groups of an alkoxy silane compound. Below 0 \*\*, advance of a reaction has dramatically late reaction temperature, and there is a danger that control of a reaction cannot be performed, but it will gel or particles will become a size unsuitable to this invention and form, above 200 \*\* impractically.

The alkoxy silane compound used at this time is a compound shown with a following general formula (1), or its partial condensate.

[http://www4.ipdl.inpit.go.jp/cgi-bin/tran\\_web.cgi\\_ejie?atw\\_u=http%3A%2F%2Fwww4.ipdl.inpit.go...](http://www4.ipdl.inpit.go.jp/cgi-bin/tran_web.cgi_ejie?atw_u=http%3A%2F%2Fwww4.ipdl.inpit.go...) 2010/10/22

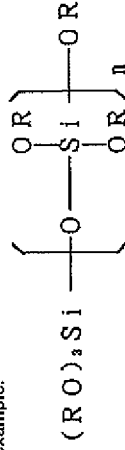
$(\text{CH}_3)_n\text{Si}(\text{OR}^1)_{4-n} \quad (1)$

(However,  $\text{R}^1$  in a formula shows an alkyl group, an aryl group, an alkenyl group, or a hydrogen atom, and  $n$  is an integer of 0-1.)

With the alkyl group, aryl group, and alkenyl group which are substituent  $\text{R}^1$  in the above-mentioned general formula (1), a methyl group, phenyl groups, such as an ethyl group, *n*-propyl group, *n*-butyl group, *i*-butyl group, *s*-butyl group, and *t*-butyl group, a tolyl group, a mesityl group, etc.; there are a vinyl group, 1-propenyl group, an allyl group, an *i*-propenyl group, etc.

Specifically A tetramethoxy silane, a tetraethoxysilane, tetra-*n*-propoxysilane, Tetra-*i*-propoxysilane, tetra-*n*-butoxysilane, tetra-*i*-butoxysilane, Tetra-*t*-butoxysilane, methyl trimetoxysilane, methyl triethoxysilane, methyltri-*n*-propoxysilane, methyltri-*i*-propoxysilane, methyltri-*n*-butoxysilane, methyltri-*i*-butoxysilane, etc. are mentioned.

The above alkoxy silane monomer compounds carry out dehydration condensation, and serve as oligomer, such as a dimer and a trimer, and such partial condensate is shown by the following formulas as an example.



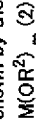
(但  $n$  は整数)

That is, it is used as an alkoxy silane compound which will serve as a raw material of the compound silica which is one of the constituents of this invention if it is a thing of the reaction mechanism which generates a silanol group and alcohol by a hydrolysis reaction from the alkoxy group in a silane compound, and \*\*\*\*. Use for a reaction and as a hanging-down basic catalyst Ammonia, ethylamine, Although solid bases, such as inorganic bases, such as amines, such as diethylamine and triethylamine, sodium hydroxide, and a potassium hydrate, anion exchange resin, lead hydroxyapatite, a hydrotalcite, a bismuth trioxide, and a hydrous bismuth oxide (V), etc. can be used. Low boiling amine and especially ammonia are preferred. It will not be limited especially if there are water and alkoxy silane, and compatibility as a solvent. As this solvent, alcohols, such as methanol, ethanol, propanol, and butanol, Although glycol mono- ether, such as ketone, such as acetone and methyl ethyl ketone, ethylene glycol methyl ether, ethylene glycol ethyl ether, ethylene glycol butyl ether, and propylene glycol ethyl ether, is mentioned, it is preferred to use a kind or two sorts or more of mixed solvents chosen from the ketone which shows good solvent power to the epoxy resin and polyvinyl butyral resin which are the components of this invention, and glycol mono- ether.

making concentration of the alkoxy silane at the time of making it react into oxide conversion -- 0.01-0.5mol/\*\*\*\* -- they are 0.03-0.3mol/\*\*\*\* preferably. The grain child generation which is low concentration becomes very slow from 0.01 mol / \*\*\*\*, and there is fear of the gelling by it being high concentration from 0.5 mol / \*\*\*\*.

When manufacturing a multiple oxide or compound hydroxide with zirconia, titanium, aluminum, boron, etc., it is obtained by carrying out the addition reaction of the metal alkoxide or metal coordination compound of this metal after coexistence or silica particle generation, when an alkoxy silane compound is hydrolyzed. In that case, the various multiple oxides or compound hydroxide of a form can be obtained by choosing the addition method and a reaction condition suitably.

The metal alkoxide or metal coordination compound which can be used at this time will not be limited especially if it has the character hydrolyzed under basic conditions, but its metal coordination compound shown by the metal alkoxide shown with a following general formula (2), (3), or (4) is especially preferred.



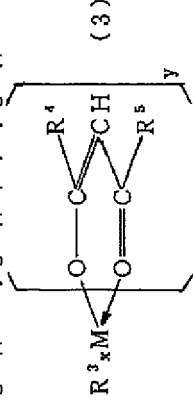
(However, in the inside M of a formula, the metal atom of  $m$  value is shown,  $\text{R}^2$  shows an alkyl group, an aryl group, an alkenyl group, or a hydrogen atom, and  $m$  shows the integer of 2-4)

The metal M in the above-mentioned general formula (2) is 2 - tetravalent metal, and titanium, a zirconium, aluminum, boron, etc. are mentioned.

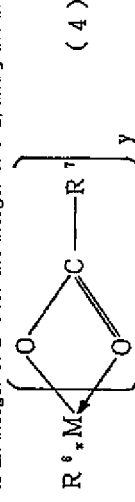
The alkyl group, aryl group, alkenyl group which are substituent  $\text{R}^2$ , if it carries out, they are; vinyl group and 1-propenyl groups, such as; phenyl groups, such as a methyl group, an ethyl group, *n*-propyl group, *i*-propyl group, *n*-butyl group, *i*-butyl group, *s*-butyl group, and *t*-butyl group, a tolyl group, and a mesityl

[http://www4.ipdl.inpit.go.jp/cgi-bin/tran\\_web.cgi\\_ejie?atw\\_u=http%3A%2F%2Fwww4.ipdl.inpit.go...](http://www4.ipdl.inpit.go.jp/cgi-bin/tran_web.cgi_ejie?atw_u=http%3A%2F%2Fwww4.ipdl.inpit.go...) 2010/10/22

group, an allyl group, *i*-propenyl group, etc.



(However, in the inside M of a formula,  $\text{R}^3$  shows an alkoxy group,  $\text{R}^4$  and  $\text{R}^5$  show an alkyl group, an aryl group, or an alkoxy group, an  $n$ -valent ( $n$  is an integer of 2-4) metal atom is shown, and  $x+y$  shows [ $y$  of  $x$  is an integer of 2-4 for the integer of 0-2, and] the integer which is  $n$ )



(However, in the inside M of a formula,  $\text{R}^6$  shows an alkoxy group,  $\text{R}^7$  shows an alkyl group, an aryl group, or a hydrogen atom, an  $n$ -valent ( $n$  is an integer of 2-4) metal atom is shown, and  $x+y$  shows [ $x$  is an integer of 0-3 and] the integer which is  $n$ .)

The metal M of the metal coordination compound shown by the above (3) and (4) formulas is 2 -

tetravalent metal, for example, can mention titanium, a zirconium, aluminum, boron, etc. Substituent  $\text{R}^3$  and  $\text{R}^6$  are alkoxy groups, such as a methoxy group, an ethoxy group, *n*-propoxy group, and *i*-propoxy group.

$\text{R}^4$  and  $\text{R}^5$  A methyl group, an ethyl group, *n*-propyl group, *n*-propyl group, such as alkyl-group; phenyl groups, such as *i*-propyl group, *n*-butyl group, *i*-butyl group, and *s*-butyl group, a tolyl group, and a mesityl group; they are an alkoxy group or hydrogen atoms, such as a methoxy group, an ethoxy group, *n*-propoxy group, and *i*-propoxy group.

$\text{R}^7$  is aryl groups, such as alkyl-group; phenyl groups, such as a methyl group, an ethyl group, *n*-propyl group, *i*-propyl group, *n*-butyl group, *i*-butyl group, and *s*-butyl group, a tolyl group, and a mesityl group, or a hydrogen atom.

Specifically Tetramethoxy titanium, tetraethoxytitanium, tetra-*n*-propoxytitanium, Tetra-*i*-propoxytitanium, tetra-*n*-butoxytitanium, tetra-*i*-butoxytitanium, Tetra-*t*-butoxytitanium, a diacetyl acetate CHITAMPROPI rate, Titanium tetrakis (acetylacetate), a tetraethoxyzirconium, A tetra-*i*-propoxyzirconium, a tetra-*n*-propoxyzirconium, Tetra-*n*-butoxyzirconium, tetra-*i*-butoxyzirconium, Tetra-*t*-butoxyzirconium, a zirconium screw (acetylacetate), Zirconium tetrakis (acetylacetate), bird ethoxyaluminum, Tri-*n*-propoxyaluminum, tri-*i*-propoxyaluminum, tri-*t*-butoxyaluminum, diethyl acetate aluminum JISOPUROPIRETO, aluminum tris (ethylacetate), boric acid triethyl borate, etc. are raised.

O Solvent it is common to make this invention constituent into solution form, and to coat, and the effect outstanding by using it for a solvent, dissolving, without being is demonstrated. As a solvent used, for example Toluene, xylene, ethylbenzene, Aromatic solvents, such as mesitylene; Acetone, methyl ethyl ketone, methyl isobutyl ketone, Mono- ketones, such as dibutyl ketone, cyclohexanone, and isophorone; An acetylacetone, 1,3-diketone system solvents and beta-ketocarboxylic acid ester solvents, such as methyl acetate and ethyl acetate; Methyl alcohol, isopropyl alcohol, isobutyl alcohol, isobutyl alcohol, Alcohols solvents, such as *n*-butyl alcohol, 3-methoxy butyl alcohol, and 3-methyl-3-methoxy butyl alcohol; Ethylene Glycol methyl ether, Ethylene glycol ethyl ether, ethylene glycol butyl ether, Diethylene glycol ethyl ether, propylene glycol methyl ether, Glycol mono- ethers solvents, such as propylene glycol ethyl ether and propylene glycol butyl ether, Ethyl acetate, Butyl acetate, ethylene glycol methyl ether acetate, propylene glycol-methyl ether acetate, propylene glycol ethyl ether acetate, adipic acid dimethyl ester, Ester solvents, such as 2-ethoxyethyl propionate, etc. are mentioned. In these solvents, two or more sorts of partially aromatic solvents chosen from the ketone system, aromatic system, and glycol ether system which have good solvent power are desirable still more preferred, the boiling point in ordinary pressure has a moderate volatilization rate, and not less than 100 \*\* a not less than 200 \*\* solvent has it

[http://www4.ipdl.inpit.go.jp/cgi-bin/tran\\_web.cgi\\_ejie?atw\\_u=http%3A%2F%2Fwww4.ipdl.inpit.go...](http://www4.ipdl.inpit.go.jp/cgi-bin/tran_web.cgi_ejie?atw_u=http%3A%2F%2Fwww4.ipdl.inpit.go...) 2010/10/22

in respect of drying property. [ preferred ]

O Additive agent It is possible to blend the additive agent suitable for the various purpose for this invention constituent. That by which the object for prizes is widely carried out in the field concerned as this additive agent can be mentioned, and the following can be mentioned.

Carbon black, acetylene black, rum black, boneblack, Black pigments, such as black iron oxide, White pigment, zinc dichromate, such as titanium oxide, a flower of zinc, and lead white, rust preventive pigment [ such as strontium dichromate, ], --- metal powder [ such as the end of extender, zinc dust, such as calcium carbonate, talc, clay, kaolin alumina, and a silicic acid anhydride, and an aluminium powder, ], --- in addition to this --- paints, such as molybdenum disulfide, tetrafluoroethylene powder, and vinylidene fluoride powder.

In addition, various additive agents, such as a viscosity modifier, \*\*\*\*\*, a leveling agent, a siloxane bond condensation catalyst, an antioxidant, and an ultraviolet ray absorbent, may be added. This additive agent can be blended in the suitable stage at the time of manufacture.

O Blending ratio 90 / 10 - 40/60 have preferred C/B at a weight ratio, and, as for the blending ratio of the organic silicon compound (A ingredient is called below) of this invention constituent, an epoxy resin (B ingredient is called below), and polyvinyl butyral resin (C ingredient is called below), 85 / 15 - 50/50 are more preferred. If it separates from this rate, the adhesion to the base material of resin will fall, or the adhesion at the time of processing is inferior, or film formability falls on the occasion of the coating to a coating object further. 5 / 95 - 70/30 have preferred A/(B+C) at a weight ratio, and, as for the blending ratio of A ingredient, 10 / 90 - 80/40 are more preferred. The coat from which A/(B+C) was obtained when less than 5/95 has low crosslinking density, and it becomes weak, or hardness becomes low. When A/(B+C) exceeds 70/30, film formability falls and a precise film is hard to be obtained.

It is desirable still more preferred that it is 1 / 100 - 200/100 in a weight ratio to the total quantity of an organic silicon compound, an epoxy resin, and butyral resin, and the rates of a silica particle are 5 / 100 - 150/100. Since the blending effect of paints will not be acquired or film strength will fall remarkably if it separates from this rate, it is not desirable.

As for the concentration of a nonvolatile matter, 3 to 90 weight % is preferred, and it is 10 to 60 weight % more preferably. If it separates from this rate, the liquid viscosity at the time of a coating will become less suitable, and will produce fault in the coat after a coating.

O Adjustment method Although the preparing method in particular of this invention constituent should not be limited, For example, after making an epoxy resin and polyvinyl butyral resin dissolve in a solvent, blending an organic silicon compound and agitating under ordinary temperature or heating, the method of adding a silica particle and making it mix uniformly is preferred in respect of the storage stability of liquid. O Application base material Although this invention constituent may be widely applied to a metal base, iron and an iron alloy, aluminum and an aluminum alloy, copper, and a copper alloy. The object for prizes is carried out to the base material with which various plating of galvanization and zinc alloy plating, a nickel plate, chrome plating, a cadmium plate, etc. was performed. The galvanization especially given by methods, such as electroplating and hot dipping, nickel and galvanization, Zinc, such as iron and galvanization, tin, galvanization, zinc alloy plating, and the rust prevention performance chromate treatment and phosphate treating excelled [ rust prevention performance ] in this plated surface to a \*\*\*\*\* steel plate or steel materials are demonstrated.

O The application method The solution form this invention constituent can be easily applied to a metal base, and can apply publicly known coating methods, such as a spray coat, dip coating, a roll coat, and brush coating, as the application method. Under after [ a coating ] ordinary temperature, or a heating condition, by removing a solvent, a uniform coat is formed and the purpose of this invention can be attained.

As thickness, 0.1-100g / m<sup>2</sup> is preferred, and more preferably 0.3-30g / m<sup>2</sup>. Especially desirable things are 0.5-10g / m<sup>2</sup>, and especially the thing for which rust prevention can be remarkably given by that cause even if it is the thickness below 10g / m<sup>2</sup> is the big feature of this invention constituent. Although the metal which processed this invention constituent can be used in a form as it is, it is also still more possible to carry out finish coating of the various paints, such as a solvent system, a drainage system, a non-solvent system, and a granular material system. Methods, such as a spray coat, a roll coat, dip coating, electrostatic coating, and electropainting, can be used as a coating method, and it dries and hardens by methods, such as room temperature setting, hot wind heating, high frequency induction heating, far infrared heating, UV irradiation, and electron beam irradiation. a steel pipe/paint used with electropainting / middle coat / finishing in the field of finishing used as an example of this paint in the field

[http://www4.ipdl.inpit.go.jp/cgi-bin/tran\\_web.cgi\\_ejie?atw\\_u=http%3A%2F%2Fwww4.ipdl.inpit.go...](http://www4.ipdl.inpit.go.jp/cgi-bin/tran_web.cgi_ejie?atw_u=http%3A%2F%2Fwww4.ipdl.inpit.go...) 2010/10/22

of the under coat/finishing of the precoat steel plate appointed to a position of a trust in the field of building materials, a household appliance, etc., a household appliance, etc., a car, etc., and engineering works and construction are mentioned.

# "OPERATION"

this invention constituent is attained by carrying out combination distribution of the silica particle to the constituent which consists of an organic silicon compound, an epoxy resin, and polyvinyl butyral resin. The coat performance which was excellent when it coated to metal and the galvanized steel sheet in which a zinc system galvanized steel sheet, and chromate treatment and phosphate treating were performed especially is demonstrated.

The Reason for demonstrating the coat performance excellent in this invention constituent is presumed as follows, although it is not clear.

\*\* In order for the activity SiOH to remain in the compound silica surface which deposits at the time of hydrolysis, and for the reaction of the SiOH group and the OH radical in resin to occur, to make the ingredient highly decoded to a resinous principle and silica components from basic conditions and to form a precise film after a coating.

\*\* In order to form the coat which the compound silica which deposits is uniform particle diameter, was moreover uniformly distributed at the time of hydrolysis, and was uniformly distributed after the coating on basic conditions.

\*\* Firm adhesion is revealed by reactant high metallic components other than Si reacting to the OH radical in resin, and performing the metal or the chemical conversion ingredient, the reaction, or a chemical combination of a ground.

\*\* The activity OH radical of the above-mentioned compound silica reacts to the resinous principle of finish coating, and forms a firm combination.

Hereafter, an embodiment is given and described concretely.

"An embodiment and a comparative example"

The synthetic example compound silica A dropping funnel of compound silica, a thermometer, and an agitating device to the reaction vessel which it had 80 copies of tetraethoxysilanes. After teaching 100 copies of isopropanols and carrying out temperature up to 70 \*\*, 0.5 copy of ethylamine, 15.3 copies of pure water, and the mixed liquor of 40 copies of isopropanols were dropped gradually, it was made to react at 70 \*\* as it is for 3 hours, and the nebula liquid in which the silica particle with a mean particle diameter of 0.1 micrometer deposited was obtained. It condensed under decompression of this nebula liquid, and the heating residue was adjusted to 10%. This heating residue took the nebula liquid 1g to the aluminum cup, and at 150 \*\* it was heated for 20 minutes and it asked for it as that survival rate.

A compound silica B dropping funnel, a thermometer, and an agitating device to the reaction vessel which it had 60 copies of tetraethoxy silanes. After preparing 110 copies of propylene glycol monoethyl ether and carrying out temperature up to 40 \*\*, The mixed liquor of 0.5 copy of ethylamine, 9.2 copies of pure water, and 30 copies of propylene glycol monoethyl ether was dropped gradually, it was made to react at 40 \*\* as it is for 3 hours, and the fine nebula liquid in which the silica particle with a mean particle diameter of 0.02 micrometer deposited was obtained. It condensed under decompression of this nebula liquid, and the heating residue was adjusted to 10%.

A compound silica C dropping funnel, a thermometer, and an agitating device to the reaction vessel which it had 60 copies of tetraethoxysilanes, 19.8 copies of zirconium tetrakis (acetylacetonate) and 130 copies of methyl ethyl ketone are taught. After carrying out temperature up to 70 \*\*, 0.3 copy of ethylamine and the mixed liquor of 20 copies of 10.0 copies of pure water methyl ethyl ketone were dropped gradually, it was made to react at 70 \*\* as it is for 4 hours, and the fine nebula liquid in which compound silica with a mean particle diameter of 0.05 micrometer deposited was obtained. It condensed under decompression of this nebula liquid, and the heating residue was adjusted to 10%.

the reaction vessel provided with the compound silica D dropping funnel, the thermometer, and the agitating device --- the partial condensate (the Tama Chemicals Co., Ltd. make.) of a tetraethoxysilane After preparing 40 "40 copies of trade name" ethyl silicate, and 100 copies of propylene glycol monoethyl ether and carrying out temperature up to 70 \*\*, 0.3 copy of ethylamine, 10.0 copies of pure water, and the nebula liquid in which trickled gradually the mixed liquor of 25.0 copies of propylene glycol monoethyl ether, it was made to react at 70 \*\* as it is for 3 hours, and the silica particle deposited with ten copies of was gradually dropped at the liquid which diluted 2.8 copies of tetra-propoxytitanium with ten copies of propylene glycol monoethyl ether in the obtained liquid, and was made to react at 70 \*\* for 2 hours, and the nebula liquid in which compound silica with a mean particle diameter of 0.1 micrometer deposited was obtained. It condensed under decompression of this nebula liquid, and the heating residue was adjusted to

[http://www4.ipdl.inpit.go.jp/cgi-bin/tran\\_web.cgi\\_ejie?atw\\_u=http%3A%2F%2Fwww4.ipdl.inpit.go...](http://www4.ipdl.inpit.go.jp/cgi-bin/tran_web.cgi_ejie?atw_u=http%3A%2F%2Fwww4.ipdl.inpit.go...) 2010/10/22

10%.

Embodiment 1 In the flask which it had, an agitator Epicoat 1004 (product made from Oil recovery Shell Epoxy) 60 copy, S Iek SL1 (made by Sekisui Chemical Co., Ltd.) 20 copy, compound silica A200 copy, After teaching 330 copies of propylene glycol monomethyl ether, and 40 copies of xylene and agitating for 3 hours, add 20 copies of gamma-aminopropyl triethoxysilane (molecular weight 221), and further as a catalyst. After adding one copy of dibutyltin dilaurate, the mixture solution was carried out and the homogeneous solution was obtained.

The following examinations were presented with this constituent.

O Creation of a test panel First, to the galvanization electrolytic chromate treatment board (70x150x0.475mmt), this solution was applied to 1g of thickness / m<sup>2</sup>, and was heat-treated by the bar coating machine, and the test panel (20 \*\* of highest attainment board temperature x 60 seconds) was obtained.

The characteristic measurement examination was done as follows.

O Corrosion resistance test It carried out for neutral salt spray test (JIS-Z-2317) 240 hours, after putting a cross out into a test panel. This result is summarized in Table 1. Although the examination of unapplied elegance was also done as comparison, it was generated by rust after 240-hour progress. The standard of evaluation was carried out as follows.

O : Rust-generating-less \*\*: White rust 2-mm or less x : white rust [ ] -- not less than 2 mm again -- as the corrosion resistance test after processing -- a test panel -- Erichsen 7mm -- extruding (JIS-K-5400) -- it carried out for neutral salt spray test (JIS-Z-2371) 240 hours, after carrying out. This result was summarized in Table 1. The standard of evaluation was carried out as follows.

O : Rust-rusting-less \*\*: x with [ less than 10% of ] white rust of processing section area : Not less than 10% of O adhesion test with white rust of processing section area The adhesion test examined primary adhesion and secondary adhesion. By sticking and exfoliating, a primary adhesion test minces the grid of 100 pieces at intervals of 1 mm to each test panel film surface, performs it to this grid, and adhesive tape (pure water) for 240 hours, and the grid of the 1-mm interval same within 30 minutes as the above was minced after that, and adhesive tape was performed to this grid by sticking and exfoliating. Finishing adhesion examined primary adhesion and secondary adhesion for Kansai Paint Co., Ltd. make Amylac #805 white in a similar manner after 40micro paint to each test panel. These results were summarized in Table 1.

O : Peeling-less \*\*: It peels and is 10% or less x : It peeled, compound silica A-D was mixed at a rate of Table 1 by the same method as the more than 10% embodiment 2-6 embodiment 1, and the homogeneous solution was obtained. Using these constituents, the corrosion resistance test and the adhesion test were done like Embodiment 1, and these results were summarized in Table 1.

Embodiment 7 The compound silica A was mixed at a rate of Table 1 by the same method as Embodiment 1, and the homogeneous solution was obtained. Using this presentation part, the corrosion resistance test and the adhesion test were done like Embodiment 1, and summarized that result in Table 1.

Embodiment 8 Aerosil 200 (Japanese Aerosil, Inc.) was mixed at a rate of Table 1 by the same method as Embodiment 1, and the homogeneous solution was obtained. Using this constituent, the corrosion resistance test and the adhesion test were done like Embodiment 1, and summarized these results in Table 1.

Comparative example 1 Although the same method as Embodiment 1 was taken, it carried out without blending compound silica and the homogeneous solution was obtained. Using this constituent, the corrosion resistance test and the adhesion test were done like Embodiment 1, and summarized these results in Table 1.

Comparative example 2 The aluminum oxide C (Japanese Aerosil, Inc.) was mixed at a rate of Table 1 by the same method as Embodiment 1, and the homogeneous solution was obtained. Using this constituent, the corrosion resistance test and the adhesion test were done like Embodiment 1, and summarized these results in Table 1.

表

No	シリカ粒子の種類	添加割合	付着量 g/m <sup>2</sup>	耐食性		密着性		密着性 (上塗り)	
				クロスカット	エリクセン	初期密着性	温床密着性	初期密着性	温床密着性
本発明例	1 複合シリカA	100/20	1	○	○	○	○	○	○
	2 複合シリカB	100/20	1	○	○	○	○	○	○
	3 複合シリカC	100/20	1	○	○	○	○	○	○
	4 複合シリカD	100/20	1	○	○	○	○	○	○
	5 複合シリカA	100/5	1	△	○	○	○	○	○
	6 複合シリカA	100/200	1	○	△	○	△	○	△
	7 複合シリカA	100/20	5	○	○	○	○	○	○
	8 アエロジル200	100/20	1	○	△	○	△	○	x
比較例	1 配合無し	100/0	1	x	x	○	x	○	x
	2 アルミニウムオキシサイドc	100/20	1	x	x	○	x	○	x

(\*\*). A metal base, when it applies [ this invention ] to a galvanization system steel plate especially, even if it is the very thin thickness below 5g / m<sup>2</sup>, it excels in good processability and the rust prevention of a processing section, and it is transparent, and since a coat has the adhesion outstanding also as a paint ground at the time of the resistance over the remains of a fingerprint and abrasion at the time of handling being also strong, and painting it on it variously, in various industries, it contributes as rust prevention treatment after the chemical conversion of a steel plate widely.

[Translation done.]

(51) Int.Cl.<sup>8</sup>

識別記号

F I

C 0 9 D 5/08

C 0 9 D 5/08

5/00

5/00

D

129/14

129/14

163/00

163/00

請求項の数1 (全 8 頁)

(21) 出願番号 特願平2-334981

(22) 出願日 平成2年(1990)11月30日

(65) 公開番号 特開平4-202481

(43) 公開日 平成4年(1992)7月23日

審査請求日 平成8年(1996)12月25日

(73) 特許権者 999999999

東亜合成株式会社

東京都港区西新橋1丁目14番1号

(72) 発明者 吉見 道成

愛知県名古屋市港区船見町1番地ノ1

東亜合成化学工業株式会社名古屋総合研  
究所内

(72) 発明者 俵 邦夫

愛知県名古屋市港区船見町1番地ノ1

東亜合成化学工業株式会社名古屋総合研  
究所内

(72) 発明者 広瀬 俊良

愛知県名古屋市港区船見町1番地ノ1

東亜合成化学工業株式会社名古屋総合研  
究所内

最終頁に続く

(54) 【発明の名称】 金属表面処理用組成物

1

(57) 【特許請求の範囲】

【請求項1】 アミノアルキル基とアルコキシシリル基を有する有機ケイ素化合物、エポキシ樹脂、ポリビニルブチラール樹脂及び有機溶剤分散性シリカ粒子からなることを特徴とする金属表面処理用組成物。

【発明の詳細な説明】

(イ) 発明の目的

「産業上の利用分野」

本発明は金属表面処理用組成物に関するものであり、更に詳しくは、化成処理の施された亜鉛メッキまたは亜鉛系合金メッキ鋼板等に適用した場合に長期にわたり良好な塗膜密着性・耐傷性・防錆性を有し、かつ、塗装下地としても有効な皮膜を形成する表面処理用組成物に関するものであり、機械工業、電気機器工業、自動車工業等金属を使用する各種産業分野で広く利用できるもので

2

ある。

「従来の技術」

従来から亜鉛メッキ鋼板または亜鉛合金メッキ鋼板の表面を種々の腐食環境から保護する目的で数多くの処理剤・塗料が使用されている。その中でもリン酸塩処理またはクロメート処理が一般的である。しかしながら、それ単独では一次防錆的な役割しかなく、短時間で錆が発生したり、又、上塗りを前提とする場合でも、上塗りまでの保管期間中に発錆するなどの問題があった。

10 近年、防錆性の向上を目的として、クロメート処理やリン酸塩処理などの化成処理を施した亜鉛系メッキ鋼板上に樹脂皮膜を形成させた鋼板が提案・実用化されている。かかる鋼板には、取扱時の耐傷性、耐指紋性、成形加工時の密着性、耐亀裂性、また、上塗り塗装時の塗着性、密着性、そして、防錆性、耐水性等の耐久性等が要